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# HEATED STAGE FOR A SCANNING PROBE MICROSCOPE

## CROSS-REFERENCE TO RELATED APPLICATION

This application is a continuation-in-part of U.S. patent application Ser. No. 08/551,836, now U.S. Pat. No. 5,654, 546, entitled "A Variable Temperature Scanning Probe Microscope Based On A Peltier Device" filed 7 Nov., 1995 in the name of inventor Stuart M. Lindsay.

### BACKGROUND OF THE INVENTION

#### 1. Field of the Invention

This invention relates to scanning probe microscopy. <sup>15</sup> More particularly, the present invention relates to temperature control of samples under investigation in a scanning probe microscope.

### 2. The Prior Art

It is often desirable to control the temperature of a sample that is being studied in a scanning probe microscope (SPM) such as the scanning tunneling microscope (STM) or atomic force microscope (AFM). This is because many aspects of surface structure and chemistry are sensitive to temperature, so variable temperature operation adds greatly to the utility of the scanning probe microscope.

Scanning probe microscopes have been constructed which operate in a cryogenic fluid or inside a high vacuum chamber. In each case it is relatively straightforward to control the temperature of the sample (and/or the microscope as well). However, in the case of microscopes designed to operate in ambient air (or some gas at or near ambient pressure) it is more difficult to design a heated sample stage. The reason is that convection caused by hot gasses and temperature gradients across the microscope (the body of which is assumed to be at ambient temperature) causes mechanical instabilities which degrade the resolution of the microscope.

One solution to this problem is to make the heater very 40 small, comparable in size to the probe of the microscope. The heated region itself can then be raised to a high temperature with a very small heat input, with the result that the rest of the sample stage and microscope is not disturbed. Such an arrangement has been built by M. DiBattista et al., 45 and is described in "A Microfabricated Hot Stage for Scanning Probe Microscopes" (1996), using integrated-circuit manufacturing technology. This approach is illustrated in the diagram of FIG. 1. In FIG. 1 the sample stage 10 is a silicon wafer of high resistivity, onto which is patterned a pair of 50 electrical contacts 12a and 12b. A small region 14 located between contacts 12a, 12b is boron-doped so as to make it electrically conductive. Electrical current passed from contact 12a to 12b (or vice versa) will cause resistive heating in region 14 depending upon the level of boron doping in 55 region 14 and the magnitude of the electrical current applied. Using this technique, the heated region can be made as small as 100 microns or so. While excellent results have been obtained with this system, only the heater itself (or thin films applied to the heater) have been studied so far. The system is too small for routine mounting of larger easily-handled samples. Furthermore, expensive microfabrication procedures are required for fabrication of this heating system.

Another approach, described by W. J. Kulnis, Jr. et al. in "A Thermal Stage for Nanoscale Structure Studies With the 65 Scanning Force Microscope", Mat. Res. Soc. Symp. Proc. Vol. 332, pp. 105–108 (1994), uses a small Peltier thermo-

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electric device to heat the sample. Peltier thermoelectric devices use electric currents to carry heat from one side of the device to another, and usually find application as small coolers. However, because heat is actively transported across the device, the device itself serves as an excellent 'insulator'. The arrangement of the apparatus of W. K. Kulnis, Jr. et al. is shown in FIG. 2. In FIG. 2, a Peltier device 16 is glued onto an X-Y scanner 18 of a scanning probe microscope. A sample to be studied 20 is glued onto the hot side 22 of Peltier device 16, so that the whole assembly of Peltier device 16 and sample 20 is scanned under probe tip 24 of the scanning probe microscope. This arrangement is simple, however the heat applied to the sample 20 is removed from the cold side 26 of the Peltier device 16 causing thermal gradients at the scanner 18. The effect is small because much of the current applied to a Peltier device is consumed in Joule heating. However, a second limitation lies with the materials used to fabricate the Peltier device itself: the manufacturers of such devices recommend that they be run at temperatures below 60° C. to avoid damage to internal solder contacts and semiconductor elements, thus the available heating range is necessarily limited by this constraint to about 60° C. or less.

Yet another arrangement has been constructed by I. Musevic et al., in "Temperature controlled microstage for an atomic force microscope", Rev. Sci. Instrum. 67 (7), pp. 2554-2556 (July, 1996). The Musevic et al. arrangement is shown at FIG. 3. A heater assembly 28, consisting of a thin film 30 of indium-tin-oxide ("ITO") is coated onto the underside 32 of a glass slide 34, the underside 40 of which also serves as a support for a sample to be studied by a scanning probe microscope. Heat is developed in the heater assembly 28 by applying an electric current to a multicore, flexible copper wire (not shown) soldered to the ITO surface at diagonally facing corners of the ITO layer 30. The heater assembly 28 is mounted onto drops 36 of an epoxy adhesive which act as thermal insulating stand-off supports for the heater assembly 28 and hold it onto an X-Y scanner 38 of a scanning probe microscope. This arrangement is capable of heating the sample to higher temperatures than the Peltierbased heater of FIG. 2, but the entire thermal gradient must be sustained across the epoxy drops 36 and the air space between the ITO layer 30 and the scanner 38. The thermal gradient across these drops 36 results in excessive thermal drift for some applications. In addition, the inherently small size of drops 36 (typically about 2 mm in diameter) provides only very limited thermal isolation of the sample stage 40 from the rest of the microscope and thus the rest of the microscope is substantially radiatively heated when the stage is hot.

## SUMMARY OF THE INVENTION

A heater for use in heating a sample stage of a microscope such as a scanning probe microscope is bonded to a sample stage which sits on a tube of a ceramic thermal insulator which is, in turn, mounted within or part of a tube of the same material. This re-entrant design provides an increased thermal path over straight line distances between the heater and the support structure for the sample stage and thus provides excellent thermal insulation, while also maximizing the thermal stability of the system.

## OBJECTS AND ADVANTAGES OF THE INVENTION

Accordingly, it is an object and advantage of the present invention to provide a heated stage for a scanning probe microscope.